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RESEARCH AND DEVELOPMENT

MONTHLY REPORT

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JULY 1961

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LIST OF SUBJECTS COVERED

I. RESEARCH

1. Special Events
2. Biotechnology
3. Chemical and Analytical Services
4. Special Projects

II. PROCESS DEVELOPMENT

5. Nitrate Reduction
6. Pilot Plant Operations
7. Reconstituted Tobacco
8. Unit Operations
9. Special Projects

III. PRODUCT DEVELOPMENT AND QUALITY ASSURANCE

10. Cigarette Development
11. Product Development Laboratory
12. Material Development
13. Tobacco Studies
14. Flavour Development
15. Material Testing
16. Cigarette and Smoke Analysis
17. Q.A. Analytical Services
18. Special Projects

IV. MISCELLANEOUS

19. Specifications
20. Process Assurance
21. Instrumentation and Process Automation
22. Documentation and Library
23. Patents
24. Legislation

Key to Distribution:

- A. Complete Report
- B. All except Research Report
- C. All except Special Projects Research and those reports which might interfere with patent considerations.
- D. Subject No 9, 12, 15, 17, 18, 19, 21, 24
- E. Subject No 10, 12, 15, 17, 19, 21

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PROJECT TITLE : BIOTECHNOLOGY
PERIOD COVERED : JUNE 17 - JULY 16, 1981
WRITTEN BY : Schulthess-D. (DIS)

GLUCOSE : NITRATE RATIO (1)

The dilution rate of the continuous denitration strongly influences the consumption of glucose. At a dilution rate of 0.1 hr^{-1} the glucose : nitrate ratio was found to be 36.9. At a dilution rate of 0.2 hr^{-1} the same ratio was lowered to a value of 25.8.

AMMONIA CONCENTRATION IN BURLEY STRIP EXTRACTS (2)

The hypothesis was postulated that a part of the ammonia found in a Burley strip extract prepared at 80°C is due to a decomposition of organic substances at this temperature (3). In order to clarify this question, two extracts were prepared at 20°C and 80°C . The two extracts showed the same N-total, N-NH_4^+ and N-NO_3^- values.

STRIP-EXTRACT DENITRATION (1.2)

Some problems were encountered with the denitration of strip-extracts at higher dilution rates (4,5).

No positive results could be obtained by lowering the extraction temperature of Burley strips from 80°C to 20°C .

The addition of salts and trace elements in quantities as they are found in the DIFCO yeast-extract improved the capacity of the denitration slightly. The dilution rate could be raised from 0.8 hr^{-1} to 0.1 hr^{-1} . Most probably the elements Mn, Mo and Zn caused this effect.

A totally normal denitration could be achieved (using the glucose : nitrate ratio of 36.9) if the strip extract was prepared in a strip : water ratio of 1 : 15. The minimal strip : water ratio that can be tolerated in the denitration process will be determined, as well as the factors that inhibit the denitration in more concentrated extracts.

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Schulthess

DIS/jig JULY 16 1981

0000144212

PROJECT TITLE : PROTAGORAS
PERIOD COVERED : MAY 21 - JULY 14, 1981
WRITTEN BY : Bindler-G.-N. (GNB)

TOBACCO EXTRACTION (1.2)

20 g of ØS-B-TOT tobacco was extracted subsequently with water, KOH and an enzyme solution. As enzyme, pronase from Boehringer (Mannheim) was used. The amount of protein extracted in the different steps (in percent of the original protein content), as well as the extraction conditions, are presented in the table below.

	pH	liquid 200 ml each	Temperature °C	Time	Enzyme	% Protein extracted
Step 1	5.2	water	60	90	-	25
Step 2	11.8	0.2N KOH	60	90	-	22
Step 3	9.8	water	37	180	100	32

Step 2	11.8	0.2N KOH	37	180	100	55
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Step 2	5.2	water	60	90	-	14
Step 3	5.2	water	60	180	-	10

The highest amount of protein that could be extracted from this cut tobacco was 80%. Using water only, 49% of proteins were found in the extracts after 3 extractions. Thus, 31% of

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proteins were extracted due to the effect of KOH and enzyme. The extraction efficiency did not change, if the enzyme was applied at the same time as KOH or in a separate extraction step.

During all these extraction steps, the MS-B-TOT blend was mostly decomposed into dust. It was no longer possible to produce cigarettes. Thus, the future work will concentrate on uncut tobacco (burley strips).

ENZYME PRODUCTION (1.2)

Protolytic enzymes were produced with 5 different strains (3). They were partially purified and will be tested for their deoroteination capacities on tobacco.

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- 1) Bindler-G.-N., Notebook 80 08 04.
 - 2) Manqilli M.-F., Notebook 80 08 05.
 - 3) Bindler-G.-N., Monthly Report, Protagoras, May 1981.
- G. Bindler*

GNR/jiq JULY 16 1981

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PROJECT TITLE : SAVOURY
PERIOD COVERED : MAY 15 - JULY 13, 1981
WRITTEN BY : Ghiste-P. (PAG)

The purpose of Project SAVOURY is to prepare flavours which, when pyrolyzed with sheet or tobacco, give Burley type flavour characteristics.

PRODUCTION OF FLAVOURS

Four samples of reaction flavours were produced under the following conditions :

Flavour code	C-38/2 (1)	C-39/1 (2)	P-16/1(3)*	P-17/1 (4)
Initial amino-acid composition	st.hydr. HCl 6N	st.hydr. HCl 6N	st.hydr. H ₃ PO ₄ 10N	st.hydr. H ₃ PO ₄ 10N
pH of reaction	7.0	7.0	6.5	6.6
Neutralizing agent	NH ₄ OH	NH ₄ OH	-	-
Sugar used	Xylose	Xylose	glucose	glucose
Total solid content %	55	55	60	60
Final pH	5.00	4.55	4.45	5.00
Temperature of reaction (°C)	110	90	90	110
Time of reaction (hr)	140	140	140	72
Ratio (aa/sugar)	1:1	1:2	1:1	1:1
Solvent	water	water	water	water

* Trial P-16/1 was carried out in order to reproduce flavour P 13/1 (5)

EVALUATION OF FLAVOURS

The flavours are currently evaluated for the following objectives :

1. LTR sprayed with flavour should give the same flavour characteristics as an Italian Burley treated in the Burley line.
2. Greek Burley tobacco, not treated in the Burley line, but sprayed with flavour should give the same flavour characteristics as a US Burley treated in the Burley line.

Flavours P-13/1, C-35/3, C-36/1 and C-37/3 were evaluated by the RME - Leaf Department.

P-13/1 came out positive, C-37/3 was still positive, but too harsh. The other two flavours were rejected as they gave an artificial taste.

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- 1) Ghiste-P., Notebook 79120 p. 14.
- 2) Ghiste-P., Notebook 79120 p. 17.
- 3) Ghiste-P., Notebook 79120 p. 23.
- 4) Ghiste-P., Notebook 79120 p. 24
- 5) Ghiste-P., Monthly Report, March 1981, p. 3.
- 6) Ghiste-P., Minutes of Meeting held on May 27, 1981.



PAG/jia/JULY 16 1981

0000144216

PROJECT TITLE : SALAMANDER II
PERIOD COVERED : JUNE 17 - JULY 29
WRITTEN BY : Murray-M. (MUM)

EXPERIMENTAL

ISH measurements using an electrostatic smoke trap operated at 20 KV gave, for ØS-8-10T cigarettes, an ISH value of $26,7 \pm 3,9$. This may be compared to an ISH value of $24,2 \pm 1,1$ using a Cambridge filter.

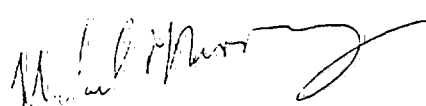
CONCLUSION

The difference is not significant. Further experiments should be carried out at higher operating voltage as the smoke precipitation at 20 KV was visibly incomplete.

FUTURE PLANS

A standard NO₂/air mixture and HPLC equipment are now available. The reaction products of NO₂ and cysteine, under test conditions, should be analysed and compared with those of the smoke vapour phase.

MUM/jiq AUGUST 3 1981



PROJECT TITLE : LAUNDRYMAN
PERIOD COVERED : JUNE 15 - JULY 29
WRITTEN BY : Piadé-J.-J. (JJP)

Cigarettes of equal RTD were made with hot water extracted ØS-B-TOT tobacco (1 hour, 60°C, 10/1 ratio) and with hot water extracted tobacco on which the concentrated extract had been resprayed.

Smoking of these sets of cigarettes versus the control for MS and SS showed that for CO :

- a) Possible structural modification by hot water extraction under the conditions used does not lead to any significant change in the CO delivery (MS -1.5% SS + 8.9%) or in the CO/TPM ratio (-2.8%).
- b) As expected, hot water extraction, when no salt is re-added, brings about significant increases in CO yields and the CO/TPM ratio is increased by 31%.

The effect of various salt casings is currently being investigated.



JJP/jud/AUGUST 3 1981

PROJECT TITLE : ROSA.
PERIOD COVERED : JUNE 17 - JULY 29
WRITTEN BY : Murray-M. (MUM)

16 single tobacco-type cigarettes made according to TLA specifications have been analysed for volatile nitrosamine delivery in sidestream smoke (1).

The results confirm the significant contribution of Burley tobacco to nitrosamine delivery.

Tobacco	mean DMN $\mu\text{g}/\text{ciq.}$	mean NPY $\mu\text{g}/\text{ciq.}$
Burley	514	201
Flue cured	226	53
Maryland	465	35
Air-cured	463	44

Filters have been prepared with varying levels of TEGDA. Cigarettes should be prepared for mainstream nitrosamine analysis whenever the backlog, due to instrumental and solvent difficulties, has been cleared up.

REFERENCE

(1) Moser-F., PME Monthly Progress Report, May 1981.

Michael J. Murray

MUM/jig AUGUST 3 1981

PROJECT TITLE : ANALYTICAL INVESTIGATIONS
PERIOD COVERED : JUNE 18 - JULY 23, 1981
WRITTEN BY : Lecoultre-E. (ETL), Moser-F. (FMO),
Murray-M. (MUM) and Piadé-J.J. (JJP)

TRIACETIN (FMO)

The two triacetin samples, one from Bergen op Zoom and the other from FIR submitted by QC (1) and extensively investigated last month (2) were fingerprinted by headspace analysis on CW 400 (column : 50 m x 0.42 mm, i.d., glass) at 50 to 110°. The peak patterns of the readily volatile fraction (bath temp. 80°) of both the Bergen op Zoom and the FIR samples were identical. However, the concentration of an unknown compound (RT 8.31 min) present in both samples was found to be higher in the sample from Bergen op Zoom than in the FIR triacetin sample (3).

A more detailed study of the particularly volatile substances in various triacetins by headspace analysis has been started.

REACTION FLAVOUR (FMO/JJP)

Work has been initiated to fingerprint the chemical composition of volatile flavour components in reaction flavour mixtures by static headspace analysis in order to assign chemical composition to subjective screening data and to check the reproducibility of the reaction flavour preparation. Five flavour samples coded P-13/1, P-15, P-16/1, P-17/1, C-38/2 and one flavour treated cigarette sample plus control were fingerprinted. Chromatograms of samples P-13/1 and P-16/1 are given in Figures 1 and 2. The initial results seem encouraging.

LC profiling of non-volatiles (sugars, Amadori compounds, etc.) in various reaction flavour samples was initiated.

CONDENSATE QUALITY CONTROL (FMO)

A simple and rapid analytical procedure for monitoring the reproducibility of the quality of dry condensate prepared for biological tests is currently being evaluated (4). Fifteen condensate samples were subjected to headspace analysis. Ten of them were prepared from the same cigarettes and were therefore identical. Based on fingerprint chromatograms all identical condensate samples were assigned correctly. A typical fingerprint chromatogram of a condensate sample is given in Fig. 3.

SUGAR ANALYSIS (JJP)

Sets of experimental cigarettes were analysed for glucose, fructose and saccharose by HPLC (5). No other sugars were detected in significant quantities.

An alternative method for glucose, fructose, saccharose, lactose, maltose and arabinose was set up to perform the same analysis by double derivatization and quantitation by gas chromatography (6) (Fig. 4). The results were found to concur well with the HPLC determinations for glucose, fructose and saccharose.

WS-14 (JJP)

GC equipment and an analytical procedure for analysing WS-14 was set up (7).

MH-30 (ETL)

The reaction products obtained by the derivatization of maleic hydrazide with dimethyl sulfate (8) were screened for possible isomers of 1-methyl-3-methoxy-pyridazone-(6). The crude reaction mixture was investigated by GC² (fused silica, 50 m x 0.3 mm, SP 2100, 150°) and GC²/MS.

The main reaction product is 1-methyl-3-methoxy-pyridazone-(6) of mp. 49-51° (yield 78-85%) (9). The isomeric 1,2-dimethyl-pyridaz-dione-(3,6) of mp. 128° (Lit. 135° (10)) is formed in ~10% yield. The compound was identified by GC²/MS (140 (M⁺, 73), 112 (15), 82 (82), 54 (37) and 43 (100) m/e) and compared with

an authentic sample prepared from sym. dimethylhydrazine dihydrochloride and maleic anhydride (10). The 3,6-dimethoxy-pyridazine isomer (mp. 108°) could not be detected and is obviously not formed under the reaction conditions used for derivatisation of maleic hydrazide.

N-NITROSAMINES (FMO/ETL)

N-Nitrosoanabasine (NAB) has definitely been detected in cigarette mainstream and sidestream smoke by GC/TEA (11) and positively identified by GC/MS by comparison with an authentic sample. NAB is separated from other tobacco specific nitrosamines on 10% SP 2100 Supelcoport 100-120 mesh (column : 2.6 m x 2 mm, i.d. glass) at 205° and 40 ml He/min (Fig. 5). Under HPLC/TEA (12) as well as GC/TEA conditions, used so far for quantitation of NNN, NATB and NNK, N-nitrosoanabasine (NAB) were eluted together with either NATB (12) or NNN. This means that values reported in the literature for NNN and NATB are probably falsified by the presence of NAB.

A sample of unknown composition submitted by the University of Neuchatel was analysed for nitrosamine content by GC/TEA and GC²/MS.

Ten tobacco extract samples from the Pilot Plant (Project LEAR) were analysed for NNN, NATB, NNK and DMN/NPY.

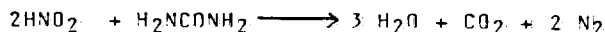
Serious problems with the GC/TEA have delayed investigations of various samples submitted by Biotechnology, Product Development and Product Research. The GC/TEA equipment is now functional.

ETNA / ONNENS (FMO)

CO, CO₂, NO, NO₂, nicotine, ammonia, humidity, temperature and particles (number and size) in exhaust gases of ETNA production were monitored during the period of July 7 to 15, 1981 (13).

NITRITE REMOVAL (MUM)

A model system was used to investigate the removal of nitrite by urea under various pH and temperature conditions (14). The results indicate a rapid and complete nitrite decomposition in the presence of urea under acidic conditions according to :



Decomposition is less rapid in citric acid than in phosphoric acid solutions. Nitrite decomposition is slightly faster at 30° than at 22°C.

Experiments with Burley strip extracts and with Burley stems confirmed the results of the model system.

Nitrite and nitrate were monitored simultaneously by HPLC on an anion exchange column with UV detection at 210 nm. Nitrate alone was measured by a specific ion electrode.

OTHERS

- Routine analyses were performed on potassium, calcium, magnesium (24 samples), phosphate, sulfate (70 samples), and alcohols (10 samples).
- CF was analysed in 20 German brands for Product Development.
- Periodic checks of the recovery of DMN in nitrosamine determinations after the clean-up procedure using ^{14}C DMN were performed.


REPORT WRITTEN

Posset-U. and Moser-F. "Potassium, Calcium and Magnesium in Fermented Tobacco Extract by Atomic Absorption", PME Analytical Method, July 1981.

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- (2) PME Monthly Progress Report, June 1981, pp. 7 - 10.
- (3) Memo from Fink-W. to Widmer-A., July 9, 1981.
- (4) Verbal request of Hackenberq-U.
- (5) Request made by the "Verband der Cigarettenindustrie" Hamburg, Germany.
- (6) Brobst-K-M. and Lott-C.E., Cereal Chemistry 43, 35 (1966) Brittain, Sullivan and Schewe, "Recent Advances in Gas Chromatography", pp. 223-229 (1971).
- (7) PME Monthly Progress Report, June 1981, p. 9.

- (8) Speck-M., PME Monthly Progress Report, June 1981.
- (9) Lecoultre-E., PME Monthly Progress Report, September 1980.
- (10) Druey-J., Meier-Kd. and Eichenberger-K., Helv. 37, 121 (1954).
- (11) Moser-F., PME Monthly Progress Report, July 1980.
- (12) Hoffmann-D. et al., Cancer Research 39, 2505 (1979).
- (13) Memo from Karbacher-P. to Moser-F., May 21, 1981.
- (14) Memos from Fink-W. to Gaisch-H., June 19 and 23, 1981.



Fink-W.

WAF/jiq August 4 1981

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Chromatogram showing detector response over time. The x-axis represents time in minutes, with major ticks at 0, 10, 20, 30, and 40. The y-axis represents detector response. Numerous peaks are visible, with the following retention times labeled: 0.79, 1.10, 1.25, 1.37, 1.42, 1.55, 1.68, 1.80, 1.92, 2.05, 2.18, 2.30, 2.42, 2.55, 2.68, 2.80, 2.92, 3.05, 3.18, 3.30, 3.42, 3.55, 3.68, 3.80, 3.92, 4.05, 4.18, 4.30, 4.42, 4.55, 4.68, 4.80, 4.92, 5.05, 5.18, 5.30, 5.42, 5.55, 5.68, 5.80, 5.92, 6.05, 6.18, 6.30, 6.42, 6.55, 6.68, 6.80, 6.92, 7.05, 7.18, 7.30, 7.42, 7.55, 7.68, 7.80, 7.92, 8.05, 8.18, 8.30, 8.42, 8.55, 8.68, 8.80, 8.92, 9.05, 9.18, 9.30, 9.42, 9.55, 9.68, 9.80, 9.92, 10.05, 10.18, 10.30, 10.42, 10.55, 10.68, 10.80, 10.92, 11.05, 11.18, 11.30, 11.42, 11.55, 11.68, 11.80, 11.92, 12.05, 12.18, 12.30, 12.42, 12.55, 12.68, 12.80, 12.92, 13.05, 13.18, 13.30, 13.42, 13.55, 13.68, 13.80, 13.92, 14.05, 14.18, 14.30, 14.42, 14.55, 14.68, 14.80, 14.92, 15.05, 15.18, 15.30, 15.42, 15.55, 15.68, 15.80, 15.92, 16.05, 16.18, 16.30, 16.42, 16.55, 16.68, 16.80, 16.92, 17.05, 17.18, 17.30, 17.42, 17.55, 17.68, 17.80, 17.92, 18.05, 18.18, 18.30, 18.42, 18.55, 18.68, 18.80, 18.92, 19.05, 19.18, 19.30, 19.42, 19.55, 19.68, 19.80, 19.92, 20.05, 20.18, 20.30, 20.42, 20.55, 20.68, 20.80, 20.92, 21.05, 21.18, 21.30, 21.42, 21.55, 21.68, 21.80, 21.92, 22.05, 22.18, 22.30, 22.42, 22.55, 22.68, 22.80, 22.92, 23.05, 23.18, 23.30, 23.42, 23.55, 23.68, 23.80, 23.92, 24.05, 24.18, 24.30, 24.42, 24.55, 24.68, 24.80, 24.92, 25.05, 25.18, 25.30, 25.42, 25.55, 25.68, 25.80, 25.92, 26.05, 26.18, 26.30, 26.42, 26.55, 26.68, 26.80, 26.92, 27.05, 27.18, 27.30, 27.42, 27.55, 27.68, 27.80, 27.92, 28.05, 28.18, 28.30, 28.42, 28.55, 28.68, 28.80, 28.92, 29.05, 29.18, 29.30, 29.42, 29.55, 29.68, 29.80, 29.92, 30.05, 30.18, 30.30, 30.42, 30.55, 30.68, 30.80, 30.92, 31.05, 31.18, 31.30, 31.42, 31.55, 31.68, 31.80, 31.92, 32.05, 32.18, 32.30, 32.42, 32.55, 32.68, 32.80, 32.92, 33.05, 33.18, 33.30, 33.42, 33.55, 33.68, 33.80, 33.92, 34.05, 34.18, 34.30, 34.42, 34.55, 34.68, 34.80, 34.92, 35.05, 35.18, 35.30, 35.42, 35.55, 35.68, 35.80, 35.92, 36.05, 36.18, 36.30, 36.42, 36.55, 36.68, 36.80, 36.92, 37.05, 37.18, 37.30, 37.42, 37.55, 37.68, 37.80, 37.92, 38.05, 38.18, 38.30, 38.42, 38.55, 38.68, 38.80, 38.92, 39.05, 39.18, 39.30, 39.42, 39.55, 39.68, 39.80, 39.92, 40.05, 40.18, 40.30, 40.42, 40.55, 40.68, 40.80, 40.92, 41.05, 41.18, 41.30, 41.42, 41.55, 41.68, 41.80, 41.92, 42.05, 42.18, 42.30, 42.42, 42.55, 42.68, 42.80, 42.92, 43.05, 43.18, 43.30, 43.42, 43.55, 43.68, 43.80, 43.92, 44.05, 44.18, 44.30, 44.42, 44.55, 44.68, 44.80, 44.92, 45.05, 45.18, 45.30, 45.42, 45.55, 45.68, 45.80, 45.92, 46.05, 46.18, 46.30, 46.42, 46.55, 46.68, 46.80, 46.92, 47.05, 47.18, 47.30, 47.42, 47.55, 47.68, 47.80, 47.92, 48.05, 48.18, 48.30, 48.42, 48.55, 48.68, 48.80, 48.92, 49.05, 49.18, 49.30, 49.42, 49.55, 49.68, 49.80, 49.92, 50.05, 50.18, 50.30, 50.42, 50.55, 50.68, 50.80, 50.92, 51.05, 51.18, 51.30, 51.42, 51.55, 51.68, 51.80, 51.92, 52.05, 52.18, 52.30, 52.42, 52.55, 52.68, 52.80, 52.92, 53.05, 53.18, 53.30, 53.42, 53.55, 53.68, 53.80, 53.92, 54.05, 54.18, 54.30, 54.42, 54.55, 54.68, 54.80, 54.92, 55.05, 55.18, 55.30, 55.42, 55.55, 55.68, 55.80, 55.92, 56.05, 56.18, 56.30, 56.42, 56.55, 56.68, 56.80, 56.92, 57.05, 57.18, 57.30, 57.42, 57.55, 57.68, 57.80, 57.92, 58.05, 58.18, 58.30, 58.42, 58.55, 58.68, 58.80, 58.92, 59.05, 59.18, 59.30, 59.42, 59.55, 59.68, 59.80, 59.92, 60.05, 60.18, 60.30, 60.42, 60.55, 60.68, 60.80, 60.92, 61.05, 61.18, 61.30, 61.42, 61.55, 61.68, 61.80, 61.92, 62.05, 62.18, 62.30, 62.42, 62.55, 62.68, 62.80, 62.92, 63.05, 63.18, 63.30, 63.42, 63.55, 63.68, 63.80, 63.92, 64.05, 64.18, 64.30, 64.42, 64.55, 64.68, 64.80, 64.92, 65.05, 65.18, 65.30, 65.42, 65.55, 65.68, 65.80, 65.92, 66.05, 66.18, 66.30, 66.42, 66.55, 66.68, 66.80, 66.92, 67.05, 67.18, 67.30, 67.42, 67.55, 67.68, 67.80, 67.92, 68.05, 68.18, 68.30, 68.42, 68.55, 68.68, 68.80, 68.92, 69.05, 69.18, 69.30, 69.42, 69.55, 69.68, 69.80, 69.92, 70.05, 70.18, 70.30, 70.42, 70.55, 70.68, 70.80, 70.92, 71.05, 71.18, 71.30, 71.42, 71.55, 71.68, 71.80, 71.92, 72.05, 72.18, 72.30, 72.42, 72.55, 72.68, 72.80, 72.92, 73.05, 73.18, 73.30, 73.42, 73.55, 73.68, 73.80

Headspace Fingerprint Chromatogram of RF Sample
P-13/1.

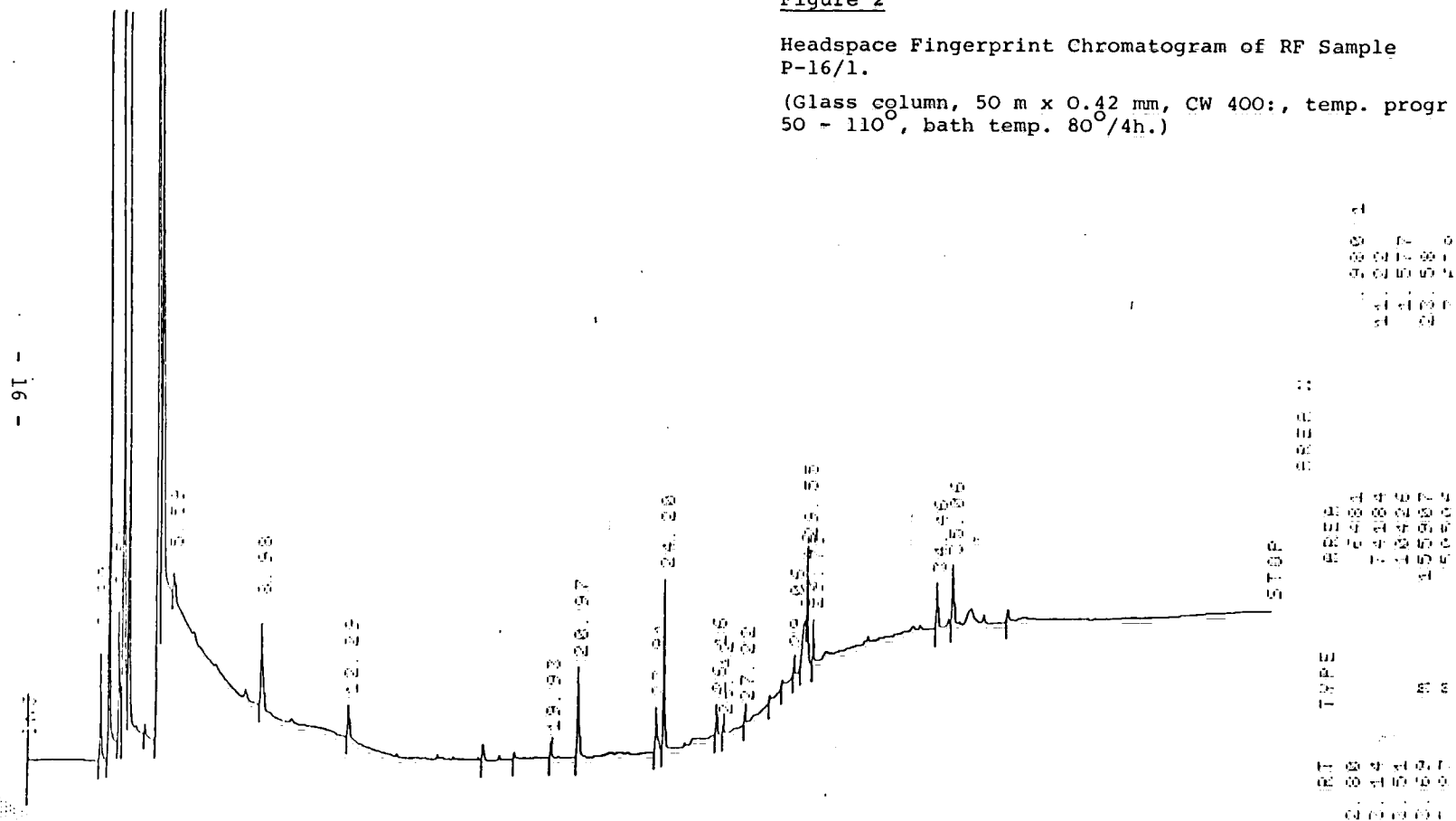
(Glass column, 50 m x 0.42 mm, CW 400; temp. progr. 50 - 110°, bath temp. 80°/4h.)

Year	1900	1901	1902	1903	1904	1905	1906	1907	1908	1909	1910	1911	1912	1913	1914	1915	1916	1917	1918	1919	1920	1921	1922	1923	1924	1925	1926	1927	1928	1929	1930	1931	1932	1933	1934	1935	1936	1937	1938	1939	1940	1941	1942	1943	1944	1945	1946	1947	1948	1949	1950	1951	1952	1953	1954	1955	1956	1957	1958	1959	1960	1961	1962	1963	1964	1965	1966	1967	1968	1969	1970	1971	1972	1973	1974	1975	1976	1977	1978	1979	1980	1981	1982	1983	1984	1985	1986	1987	1988	1989	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	2031	2032	2033	2034	2035	2036	2037	2038	2039	2040	2041	2042	2043	2044	2045	2046	2047	2048	2049	2050	2051	2052	2053	2054	2055	2056	2057	2058	2059	2060	2061	2062	2063	2064	2065	2066	2067	2068	2069	2070	2071	2072	2073	2074	2075	2076	2077	2078	2079	2080	2081	2082	2083	2084	2085	2086	2087	2088	2089	2090	2091	2092	2093	2094	2095	2096	2097	2098	2099
1900	1900	1901	1902	1903	1904	1905	1906	1907	1908	1909	1910	1911	1912	1913	1914	1915	1916	1917	1918	1919	1920	1921	1922	1923	1924	1925	1926	1927	1928	1929	1930	1931	1932	1933	1934	1935	1936	1937	1938	1939	1940	1941	1942	1943	1944	1945	1946	1947	1948	1949	1950	1951	1952	1953	1954	1955	1956	1957	1958	1959	1960	1961	1962	1963	1964	1965	1966	1967	1968	1969	1970	1971	1972	1973	1974	1975	1976	1977	1978	1979	1980	1981	1982	1983	1984	1985	1986	1987	1988	1989	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	2031	2032	2033	2034	2035	2036	2037	2038	2039	2040	2041	2042	2043	2044	2045	2046	2047	2048	2049	2050	2051	2052	2053	2054	2055	2056	2057	2058	2059	2060	2061	2062	2063	2064	2065	2066	2067	2068	2069	2070	2071	2072	2073	2074	2075	2076	2077	2078	2079	2080	2081	2082	2083	2084	2085	2086	2087	2088	2089	2090	2091	2092	2093	2094	2095	2096	2097	2098	2099

Figure 2

Headspace Fingerprint Chromatogram of RF Sample
P-16/1.

(Glass column, 50 m x 0.42 mm, CW 400:, temp. progr
50 - 110°, bath temp. 80°/4h.)



0000144326

Figure 3

Headspace Fingerprint Chromatogram
Condensate Sample.

(Glass column, 50 m x 0.42 mm, CW
progr. 50 - 110°, bath temp. 80°/

Headspace Fingerprint Chromatogram of Dry
Condensate Sample.

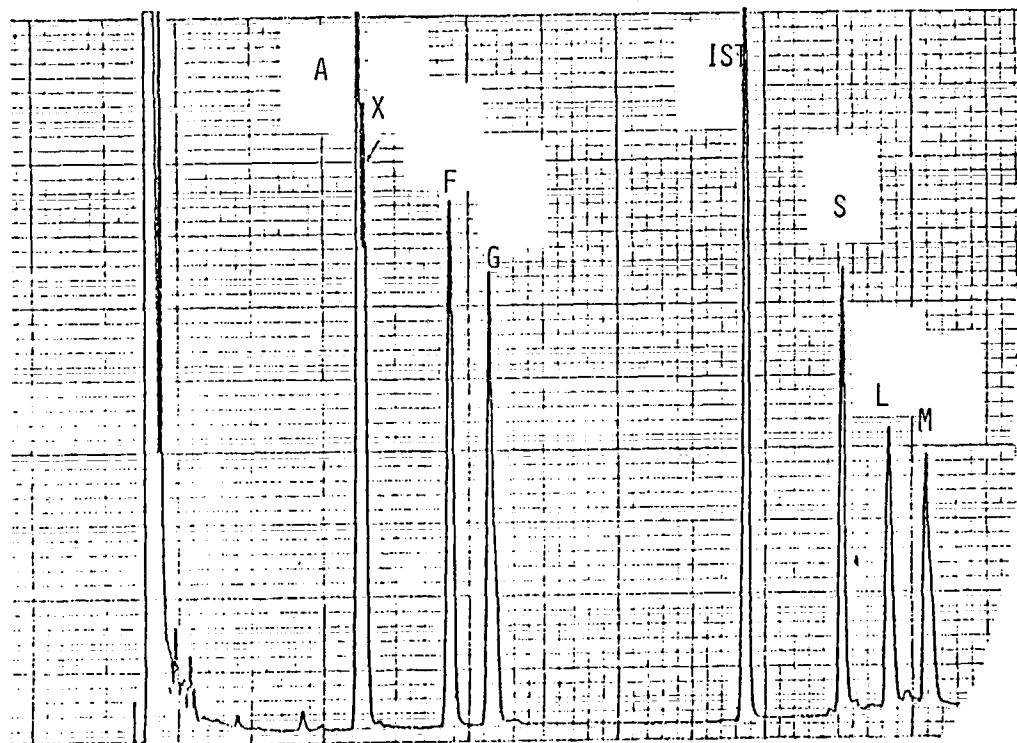
(Glass column, 50 m x 0.42 mm, CW 400; temp. progr. 50 - 110°, bath temp. 80°/2h.)

	01	02	03	04	05	06	07	08	09	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100
01	01	02	03	04	05	06	07	08	09	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100

Figure 4

Gas Chromatographic Separation of a Synthetic Mixture of Arabinose (A), Xylose (X), Fructose (F), Glucose (G), Saccharose (S), Lactose (L) and Maltose (M).

(3% SP 2250 on Supelcoport 100 - 120; Temp. Progr. 150 - 290, 6°/min.)



- 18 -

0000144228

PROJECT TITLE : NITRATE-REDUCTION BY CONTROLLED FERMENTATION
PERIOD COVERED : JUNE 21 - JULY 20
WRITTEN BY : Ruf-C. (CLR)

1. TRIALS

1.1. LEAR trial 7

Several tentatives were made to improve the denitration of strip extract during LEAR trial 7. This trial was extended to 4 weeks, and included cooling of the fermenter at each week-end (1). First of all, the pH of the extractor feedwater was lowered in order to avoid any microbial growth causing nitrite formation (2). With the continuous addition of 3% of phosphoric acid at 10% concentration, no nitrites were formed during the extraction. Later on, some trace elements (Mo, Zn, Se) were added into the fermenter but they did not improve the efficiency of the denitration, which remained uncomplete above a dilution rate of 0.1 h^{-1} . During the fourth week of the trial however, it was possible to run the process much better, after modification of the strip : water ratio of the extraction from 1 : 10 to 1 : 15. It is almost certain that a new sugar:nitrogen ratio will have to be determined for strip extracts, since an excess of sugar can have a blocking effect on the respiration of the yeasts.

1.2. LEAR trial 8

LEAR trial 8 has been running in the 20-l fermenter since July 15. Its objective is to determine the exact amount of sugar needed by the yeasts for the denitration of a strip extract. An extraction was carried out in the Rotocell with a strip : water ratio of 1 : 15 and the extract was sterilized and stored as feedstock for the 20-l fermenter (3). The sugar solution was fed separately into the fermenter by means of a metering pump which allowed accurate control of the flow-rate. The fermenter was continuously run for 110 hours at various dilution rates : 0.08, 0.14, 0.18 and 0.21 h^{-1} and total denitration was achieved all the time. The sugar:nitrogen ratio was about 24 g of 100% dextrose for 1 g of nitrogen ($\text{NH}_3\text{-N}$ and $\text{NO}_3\text{-N}$) and the acid consumption was 2% of the flow feeding the fermenter. After 22 hours at a dilution rate of 0.21, no more yeasts were found in the fermenter. They had been washed away by the dilution rate because they did not grow fast enough. However, no nitrates were detected in the extract at that time, because the denitration was probably achieved by other micro-organisms which grew faster in these conditions. At this stage, the fermentation was stopped and will be re-started with a new batch on July 21.

2. NITROSAMINES IN LEAR EXTRACTS

A series of LEAR extracts was given to the Research laboratory for analyses of nitrosamines. Although no volatile nitrosamines were detected, some relatively high concentrations of NNN, NATB and NNK were found in these extracts (4). Therefore more detailed investigations will be conducted involving samples of extracts before and after sterilization and tobaccos.

3. REFERENCES

1. Ruf-C Monthly Report, June 1981
2. Lüthi-N Monthly Report, June 1981
3. Lüthi-N Memo : Essai LEAR 8, July 14, 1981
4. Fink-W Memo to Ruf-C : Nitrosamines in LEAR, June 22, 1981

Ruf

CLR/sde/JULY 28 1981

PROJECT TITLE : PROJECT LEAR, CIGARETTE PROTOTYPES

PERIOD COVERED : JUNE 22 - JULY 23

WRITTEN BY : Borgognon-D. (DIB)

During the period of the report, the last six trial cigarettes were made according to plan, bringing the total of LEAR prototypes to 21. The tobaccos used for the following cigarettes were cut with a KTF machine.

1. Trial cigarettes

1.1. LEAR 2-17-1

This cigarette was a MLK cigarette with extracted, fermented and re-applied Burley (1 + 2).

1.2. LEAR 3-17-3

This cigarette was produced with 100% extracted, fermented and re-applied Burley. This tobacco did not undergo Burley treatment. The Burley tobacco uses a special leaf-formula selected by Mr. Karle for the LEAR Burley strip denitration (1 + 2).

1.3. LEAR 3-17-4

The cigarette was manufactured with the same tobacco as cigarette 3-17-3. The tobacco underwent Burley treatment (1 + 2).

1.4. LEAR 4-17-1

This is a MLK cigarette. The Burley tobacco was composed of 50% of extracted, fermented and re-applied Burley. The other 50% of the Burley did not undergo the same treatment, but the whole Burley tobacco underwent the Burley treatment (1 + 2).

1.5. LEAR 4-17-2

This cigarette was made with the same tobacco as cigarette 4-17-1. The Burley tobaccos have not undergone the Burley treatment (1 + 2).

1.6. LEAR 4-17-4

For this cigarette, 50% of the tobacco was extracted, fermented and re-applied Burley. The other 50% did not undergo the same treatment. The Burley treatment was applied on the whole tobacco (1 + 2).

2. Analyses

The majority of the analyses have been received and are being compiled.

3. References

1. Ruf-C "P-V de la séance de planification du projet LEAR"
(March 4 1981)
2. Borgognon-D Notebook 791104 p 47 - 49

S. Burgoyne

DIB/sde/JULY 31 1981

0000144232

PROJECT TITLE : CIGARETTE DEVELOPMENT 4
PERIOD COVERED : JUNE 20 - JULY 24 1981
WRITTEN BY : Toimil-R. (RAT)

421 HILTON ULTRA

Objective

To extend the HILTON RE family by two cigarettes having the following smoke yields :

Tar : 3 mg/cig. and 4 mg/cig.

SN : 0.3 mg/cig. and 0.4 mg/cig.

Summary

On the basis of the specifications of the HILTON ULTRA cigarette, produced in the USA, a first series of prototypes was produced.

Description of samples and results

Two series of six prototypes were produced with the objective to reach the analytical objectives.

Prototypes 3 P and 5 P satisfied the objective of 3 mg tar and prototypes 7 P and 8 P that of 4 mg tar.

Prototype		3 P	5 P	7 P	8 P
TAR	mg/cig.	3.0	3.1	4.1	3.7
SN	mg/cig.	0.24	0.25	0.32	0.32
CO	mg/cig.	7.4	6.6	7.7	6.4
NO	mg/cig.	0.13	0.12	0.14	0.14
Puff count		10.8	10.8	10.8	9.3
Dilution	%	63	64	65	52
Cigarette RTD	mm H ₂ O	111	103	104	117
Filter RTD	mm H ₂ O	116	110	113	107
Tipping paper		6 M.O.15.4.5		4 M.O.15.4.5	
Cigarette paper		WP 60	Ecusta	Mauduit	Mauduit
			708	110-6EP	120-8
Filter		Single 2.5 / 48	000 X / 100		K
Compressibility mm		2.3	2.5	2.6	2.7

Comments

The compressibility of the first two series was too high. A reduction in tobacco weight would improve the combustion and give a better impact per puff. Taste evaluation showed that the Mauduit 110-6 EP (7 P) cigarette paper is the most preferable.

Follow-up

New prototypes will be produced with different tobacco weights. This will be done in order to improve prototype 7 P and to have a cigarette with 4 mg tar which gives full taste satisfaction.

413 PALINKA

Objective

To produce the MPK brand in Hungary.

Summary

Following the taste differences of the first trial cigarettes produced with the Hungarian filters, three new filters were produced by Intertaba.

Description of samples and results

Prototype No	1 P	2 P	3 P	4 P	5 P	6 P
Blend	A	A	A	B	B	B
Filter version	I	II	III	I	II	III
TAR mg/cig.	14.8	14.7	16.0	16.0	16.0	17.0
SN mg/cig.	11.0	0.9	0.95	1.0	1.1	1.1
Puff count	9.3	9.2	9.2	9.8	9.9	9.8
Filter RTD mm H ₂ O	76	75	75	76	75	74
Cig. RTD mm H ₂ O	118	113	115	119	121	127
TA %	1.16	----	----	1.32	----	----
RS %	12.8	----	----	12.0	----	----
NO ₃ -N %	0.10	----	----	0.12	----	----
NH ₃ -N %	0.12	----	----	0.13	----	----
Filter eff. %	56	49	43	----	----	----

Comments

The different filters (versions I, II and III) are filters with different concentrations of additives for versions I and II, and version III has a less active additive.

Follow-up

On the basis of the taste evaluation of prototypes 1 P to 6 P, a decision will be taken concerning the final specifications of this project.
A product test will be organized in Hungary with the chosen version against the MAK 05 produced at Serrières.

Zouini

RAT/cap/AUGUST 5, 1981

b0000144235

PROJECT TITLE : TOBACCO STUDIES
PERIOD COVERED : JUNE 18 - JULY 27 1981
WRITTEN BY : Joseph-L. (LIJ)

TOBACCO LOT ANALYSES

Introduction of Inputs on EDP

Partial analytical results of twenty-three lots were introduced in the PME Analytical Data List.

Lots under Evaluation

Fifty-two lots.

Lots Available, but not yet Analyzed

A hundred and ten lots.

ASSISTANCE TO OTHER PROJECTS

NINO

We have received two samples of RL material. One is only the fiber support sheet (base-web), the other with the NINO denitrated extract. The analyses are under way.

Library

We have received twenty-two samples of the expansion trials made in the ETNA installation. These trials were made with different grades of Korean tobacco. The analyses are under way.

REFERENCE CIGARETTES

One reference cigarette was made this month with the HILTON ULTRA blend.

MISCELLANEOUS

Competitors' Brands

Due to a suspected change in the SELECT blend, we analyzed TLA hand-made cigarettes. Compared with the TLA cigarette made with the old SELECT blend, the first results show :

- Slightly higher TA level (1.55% instead of 1.43%)
- Higher filling power (44.5 ml/10g instead of 35.9 ml/10g)
- Lower SN filter retention
- Higher SN delivery
- Higher DPM delivery
- Slightly lower puff number
- Higher HCN delivery
- Higher aldehyde delivery

Due to the modifications of the filter and the paper porosity of the new SELECT, these changes are not reflected in the CIR analyses.

LY sept

LIJ/jud/1 AUGUST 1981

- 29 -

0000144237

PROJECT TITLE : INGREDIENT AND FLAVOUR DEVELOPMENT
PERIOD COVERED : JUNE 25 - JULY 25 1981
WRITTEN BY : FATTON J.-P (JPF)

388 / BUCA

Objective

Reduction in the ingredients in the burley casing without changing significantly the taste.

Description

Taking into consideration the comments of Panel A three new prototypes were produced.

- 10P : Is a normal Marlboro blend where only the US part of the burley tobacco was treated with the standard ingredients. The burley substitutes were added to the bright and oriental. This part of the blend received 45 liters of precutting solution instead of 40 liters to compensate for the added tobacco. The other stages of the process were standard with the same application of ingredients and the same conditions as usual.
- 11P : Is a normal Marlboro blend with standard applications except for the standard burley casing which was replaced by EBC-21.
- 12P : Is similar to version 10P but the standard burley casing was replaced by EBC-22.

These cigarettes are currently too fresh to be evaluated.

Follow-up

A Delta Panel (difference test) will be made on these prototypes. The prototypes will be submitted to Panel A for the taste evaluation.

Objective

To improve the taste and quality of the stems.

Summary

The development was made on the same flue-cured stem blend as the one used for Atlantic trials. Five different versions were submitted to Panel A.

- 1P : This version contains sfc-204 received from Richmond. This ingredient was previously foreseen to flavour the Hunter-conform RL of project Keegan.
- 2P : This version contains sfc-AC-3 (stem casing) received from Richmond.
- 3P : This version was developed at FTR and contains GAA and C14.
- 4P : This version was developed at FTR and contains TARTAR and C14.
- 5P : This version contains GAA, TARTAR and C14.

The comments of Panel A are as follows:

- Prototype 1P is the most acceptable prototype as far as a decrease in harshness is concerned, and it gives no artificial taste.

- Prototypes 3, 4 and 5 : The character of FC stems changes, the harshness doesn't decrease and the cigarettes give a strange artificial taste.

Follow-up

Small batches of stems were sprayed with:

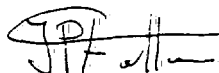
- ESTC-6 contains CW3
- ESTC-7 contains Glucose and C103
- ESTC-8 contains C14 and C103
- MF-PC

Some of these versions look promising. They will be submitted to Panel A for taste evaluation.

Contacts with suppliers

Seven samples of tobacco flavours were received from Felton (GB).

J.-P Fatton



JPF/sb/JULY 30 1981 - 32 -

0000144240

PROJECT TITLE : MATERIAL TESTING
PERIOD COVERED : JUNE 26 - JULY 28 1981
WRITTEN BY : Balliger-P (PBA)

CIGARETTE PAPER

Mauduit 325 A Verge
Wattens E 1105

Concerning the search for a second supplier, we asked Mauduit and Wattens to supply us with the above cigarette paper having the same characteristics as our standard quality, Pela 54 Mn from Schoeller & Hoesch.

As this quality is mainly being used for the diluted Muratti group, MAA-CH cigarettes produced with these two new versions are undergoing evaluation.

FILTER PAPER

Dexter USA X 4855
Dexter USA X 4902
Dexter Scotland Z 5581
Dexter Scotland Z 5644

In order to have a wider choice of filter papers suitable for the European Marlboro, Dexter proposed different qualities which were used to produce cigarette prototypes. Following subjective taste evaluation of these cigarettes, none of them were accepted.

FILTRATION MATERIAL

Amcel 3,0/ 41 000 Y

This filtration material, proposed by Amcel, corresponds almost exactly to our standard quality 3,0 / 43 000 Y from Rhodia and which is currently used in Marlboro KS (undiluted) produced by PMG-Berlin.

However, despite the satisfactory smoke yields obtained, the cigarettes produced with this new material proved to have too great a taste difference as compared with the reference cigarettes.

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TECHNICAL SHEETS

Cigarette paper

No. 30 0057 Mauduit 335 A

Filter paper

No. 23 2532 Brodano BO 0975 bis

Tipping paper

No. 32 0008 Benkert Z3/60 9696 (colour US)

No. 32 0009 Tann Z3/60 440 PE (colour US)



PBA/edk/JULY 30 1981

0000144242

PROJECT TITLE : CIGARETTE AND SMOKE ANALYSES
PERIOD COVERED : JUNE 22 - JULY 21 1981
WRITTEN BY : Senehi-F (SEF)

COMPARATIVE RESULTS OF THE OFFICIAL CANTONAL LABORATORY OF ZURICH
(DR. ROMANN) and QA FTR

A list has been established showing the tar, smoke nicotine (SN), carbon monoxide (CO) and nitrogen monoxide (NO) content of all FTR brands analysed by Dr. Romann, compared with the results obtained by QA FTR. (1)

On average, the tar and smoke nicotine results obtained by Dr. Romann are lower than those obtained by QA FTR (see table 1).

The 1981 deviations are comparable with those of 1980 for the tar but lower for the smoke nicotine:

	Deviation in %	
	<u>1981</u>	<u>1980</u>
Tar (mg/cig)	- 10.2	- 10.6
SN (mg/cig)	- 3.5	- 7.5

Compared with the printed values, all the tar and smoke nicotine results obtained by Dr. Romann are within the tolerance limits with the exception of the two following brands:

	<u>Printed values</u>	<u>Limit value</u>	<u>Dr. Romann</u>
	<u>Smoke nicotine</u>	<u>Smoke nicotine</u>	<u>Smoke nicotine</u>
Marlboro 100'S			
Red Box	1.2	1.38	1.39
Milla F	0.8	0.92	0.97

Compared with printed values, all the tar and smoke nicotine results obtained by QA FTR are within the tolerance limits.

PRODUCT REPORTS

Product reports were written on the following new or modified brands:

<u>Brand</u>	<u>Manufacturer</u>	<u>Country of sale</u>
Condor 84/F (new brand)	Brinkmann	West Germany
Lord Ultra 84/F (line extension)	Brinkmann	West Germany
Lucky Strike 84/F (new brand)	BAT	West Germany
Peer Export 84/F (new pack design)	Brinkmann	West Germany
Peter Stuyvesant Super Long 94/F (line extension)	Reemtsma	West Germany
Peter Stuyvesant Leichte Mischung 84/F (line extension)	Reemtsma	West Germany
Tobacco House No. 7 84/F (new brand)	Nestor Gianacis	West Germany

REFERENCE

1. Report Senehi-F (July 21 1981)
- Senehi*

Enclosure: Table 1

SEF/edk/JULY 30 1981

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BRANDS	TAR (15/CIG)			S N (15/CIG)			CO (15/CIG)		NO (15/CIG)	
	ROMANN	QA FTR	PRINTED VALUES	ROMANN	QA FTR	PRINTED VALUES	ROMANN	QA FTR	ROMANN	QA FTR
ARLETTE	12.5	12.8	14	0.63	0.71	0.8	17.3	17.1	0.40	0.32
BASTOS	14.9	16.1	17	0.77	0.90	1.1	14.4	15.7	0.36	0.37
BRUNETTE No. 3	1.8	2.7	3	0.17	0.25	0.3	2.2	3.4	0.06	0.06
BRUNETTE CARREE / PLATE	16.7/17.7	19.2	21	0.95/0.90	1.17	1.3	14.1/14.2	15.2	0.27/0.29	0.30
BRUNETTE DOUBLE	11.5	12.9	14	0.69	0.78	1.0	13.5	15.2	0.30	0.29
BRUNETTE EXTRA THIN / BOX	4.4/4.9	5.5	6	0.35/0.39	0.41	0.5	7.6/8.7	8.7	0.16/0.17	0.17
BRUNETTE FILTRE	15.0	14.4	16	0.91	0.93	1.1	15.9	15.6	0.25	0.29
CHAMPION	12.4	13.2	15	1.17	1.20	1.4	13.6	12.9	0.09	0.10
DIANA KING SIZE	14.3	15.7	15	0.98	1.06	1.0	15.8	17.2	0.18	0.19
FLINT SOFT	5.9	6.4	7	0.46	0.48	0.6	8.8	9.6	0.12	0.11
FLINT KING SIZE BOX	6.5	7.5	7	0.55	0.58	0.6	9.8	10.5	0.13	0.15
FLINT ULTRA	0.7	1.2	1	0.08	0.13	0.1	2.7	3.5	0.06	0.06
MARLBORO BOX	15.2	16.3	16	1.16	1.17	1.2	13.7	15.9	0.20	0.24
MARLBORO GOLD	7.1	8.8	9	0.62	0.67	0.8	9.6	11.6	0.18	0.21
MARLBORO KING SIZE	14.4	16.2	16	1.19	1.22	1.2	15.1	16.2	0.21	0.25
MARLBORO 100'S RED BOX	14.7	16.7	17	1.39	1.27	1.2	14.3	16.1	0.20	0.25
MERCEDES KING SIZE	13.8	16.5	16	0.92	1.07	1.1	16.3	16.5	0.16	0.18
MILLA	15.5	16.5	16	0.97	0.89	0.8	14.3	15.2	0.09	0.09
PS KING SIZE	15.8	17.0	18	1.32	1.28	1.2	17.2	16.6	0.34	0.36
MULTIFILTER 100's	11.1	12.5	13	1.01	0.96	1.0	12.2	13.8	0.16	0.17
MULTIFILTER KING SIZE	10.6	11.6	12	0.98	0.93	0.9	12.3	11.4	0.15	0.14
MURATTI AMBASSADOR BOX	10.0	11.8	12	0.93	0.92	0.9	11.2	12.9	0.12	0.14
MURATTI AMBASSADOR EXTRA	4.0	4.7	5	0.43	0.37	0.4	7.6	9.4	0.10	0.12
MURATTI AMBASSADOR KING SIZE	10.3	11.9	12	0.83	0.88	0.9	12.2	13.7	0.17	0.17
MURATTI 2000	5.3	6.8	7	0.46	0.52	0.6	7.9	9.6	0.12	0.12
MURATTI 2000 EXTRA LONGS BOX	5.0	7.0	9	0.47	0.58	0.8	7.0	10.7	0.10	0.12
NORRI POLE	12.7	13.8	13	0.98	0.91	0.9	13.3	14.3	0.18	0.17
PHILIP MORRIS INTERNATIONAL	15.8	16.5	17	1.17	1.11	1.1	16.2	18.1	0.20	0.27
PHILIP MORRIS KING SIZE NF	20.8	23.3	23	1.62	1.65	1.5	12.3	13.8	0.17	0.20
REGENT	13.8	15.6	15	1.05	1.03	1.0	14.4	17.6	0.18	0.19
GENERAL AVERAGE	11.11	12.37	12.74	0.84	0.87	0.91	12.12	13.27	0.18	0.19

THE SAMPLES WERE TAKEN AT ORIENTS ON MAY 21, 1981

0000144245

PROJECT TITLE : QA ANALYTICAL SERVICES
PERIOD COVERED : MAY 21 - JULY 21 1981
WRITTEN BY : Widmer-A (ALW)

1.1. METHODS

- Determination of the moisture content of tobacco. (1)
The moisture content of different samples was determined by PME method No. 210 (oven volatiles) and the "Coresta" reference method (azeotropic distillation with cyclohexane). The following results were obtained:

Sample	Distil- lation, %	O.V. (%)	Δ Dist.-O.V. (%abs.)	Δ Dist.-O.V. (% rel.)
ETNA-FC (after tower)	2.9	4.4	+ 1.5	+ 52
ETNA-FC (final product)	8.8	9.9	+ 1.1	+ 13
ETNA-FC (cut rag)	18.8	19.8	+ 1.0	+ 5.3
MLF-blend (before flavouring)	9.7	11.4	+ 1.7	+ 18
MLF-blend (after flavouring)	9.7	11.2	+ 1.5	+ 16
MAA-blend (before flavouring)	9.1	10.0	+ 0.9	+ 10
MAA-blend (after flavouring)	9.1	10.2	+ 1.1	+ 12
BRD-blend	8.7	9.3	+ 0.6	+ 6.9

The standard deviations for the two methods are similar. The results led to the following conclusions:

- The values obtained by distillation are in any case lower than the O.V. - values.
- The higher the quantity of ingredients in a blend, the higher is the difference (in relative %) between the two methods (comparison between MLF-, MAA- and BRD-blends).
- The lower the moisture content of a sample, the higher is the difference between the two methods and vice-versa. This means that in the O.V.-method, samples with a low moisture content are "over-dried" and samples with a high moisture content are not "sufficiently dried".

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1.4. ORGANISATION

- Control of ESTHER-production

Processing control of ESTHER-production has been carried out since June 1, 1981, by the QA-Laboratory in Onnens instead of by the QC-Preparation Laboratory in Serrières.

2.1. CASING KITCHEN

- BSE-AC (2)

The preparation procedure for the new solution of BSE-AC was established. Because of its strong odour, the solution must be prepared in a specially indicated tank reserved for this purpose.

- Determination of Density and Refraction in solutions (3).

The purpose of this work was to get an idea as to what extent and reliability solutions may be controlled by the determination of density and refraction. The study was made on MF-PC solutions produced in the laboratory.

Individually, each component was applied at different rates (0 %, 50 %, 80 %, 90 %, 110 %, 120 % and 150 % of the normal quantity). The final volumes of each solution (according to the standard recipe) were kept identical by completing them with water. Afterwards the density and the refraction were measured. In graphs the results were plotted against the quantity of the individual components. By introducing the standard deviation for MF-PC, obtained during the control trial (see below), the following detection limits were found:

Ingredient	in % of the standard quantity	
	by refraction	by density
Glycerine	95 - 103	97 - 105
Propylene glycol	93 - 103	70 - 130
Honey	91 - 113	93 - 105
Invert sugar	88 - 110	95 - 105
Water (final volume)	91 - 105	90 - 108

In a second step, where the same principles as those described above were applied, inversions between two similar components were examined.

Ingredient	theoretical rate(%)	detection limits as % of the theoretical rate	
		by refraction	by density
Glycerine	55	38 - 72	53 - 58
Propylene glycol	45	28 - 62	42 - 47
Honey	48	16 - 79	26 - 69
Invert sugar	52	21 - 84	31 - 74

In conclusion, even though with other solutions the composition and the interferences may be different, we can say that:

- Important errors in the quantities of the main components can be detected by combining the two methods.
- Errors in the addition of low quantity components cannot be detected (eg artificial honey flavour).
- Inversion of similar components can easily be detected in the case of humectants, but not in the case of honey and invert sugar.

- Quality Control of solutions (4)

Density and refraction values were determined systematically in all the solutions produced in the casing kitchen during two trials carried out over a period of about 3 months. The statistical evaluation of the results led to the following conclusions:

- Both shifts work identically and follow the fabrication procedures.
- Both shifts are at the same level as regards quality of solutions produced.

On the basis of the results obtained, specifications for the most important solutions (BURLEY CASING, BURLEY TOP FLAVOUR, MF-PC, etc.) were established. Furthermore, measures to improve quality, to eliminate potential danger of errors and to facilitate manipulation and working facilities were proposed and discussed with production staff (5).

3.2.1. TRIALS WITH NEW SUPPLIES OF TOBACCO INGREDIENTS

- Invert sugar (SCHOLTEN/AVEBE, Foxhol (NL);
"TRICOLIN Z 3833" / PMH Bergen op Zoom) (6)

The sample is within specifications.

- Licorice extract (MAC ANDREWS & FORBES, Croydon (USA);
produced in IRAN/yellow sheet 5923, 50 kg)
First industrial trial (MLF-CH)

Laboratory: The sample is slightly too high as regards
the content of glycyrrhizic acid.

Smoke analyses: No significant difference between the
standard and the trial was found.

Subjective evaluation: A significant difference between
the standard and the trial was found. The trial was re-
jected.

Concluded on June 15, 1981

- Licorice extract (FERTILIZER & CHEMICALS, Haifa (Israel)/
yellow sheet 6000, 50 kg)
First industrial trial (MLF-CH)

Laboratory: The sample is within the specifications.

Smoke analyses: No significant difference between the
standard and the trial was found.

Subjective evaluation: A significant difference between
the standard and the trial was found. The trial was re-
jected.

Concluded on June 15, 1981

- Licorice extract (PAULIN PAUL Ltd., London; produced in
CHINA (TIENTSIN)) (7)

The content of ash is too high. In addition to this, a
significant difference in taste and odour, compared with
the standard, was detected.

- Raw cane sugar (A. TOEPFER & CO, Hamburg; produced on
FIDJI-ISLANDS/yellow sheet 6030, 30 kg)
First industrial trial (MLF-CH)

Laboratory: The sample is within the specifications.

Smoke analyses: No significant difference between the
standard and the trial was found.

Subjective evaluation: A significant difference between the
standard and the trial was found. The trial was rejected.

Concluded on June 15, 1981

- 7
- Raw cane sugar (A. TOEPFER & CO, Hamburg; produced in JAMAICA) (8)

The sample is within the specifications.

- Raw cane sugar (A. TOEPFER & CO, Hamburg; produced in TRINIDAD) (8)

The sample is within the specifications.

- Sodium benzoate (C.D.f. CHIMIE, Paris) (9)

The sample is slightly out of specifications as regards the water content.

- Tobacco Flavour 15.96.0242 (IFF AG, Reinach (CH)/yellow sheet 6078, 120 kg)

The product was accepted as regards subjective evaluation (Panel A) of the first production of BSE and BSH.

3.2.2. QUALITY CONTROL OF TOBACCO INGREDIENTS

- Ingredients used in LECCE for DIANA-production (10)

In a trial in February 1981 it was found that the cocoa powder and the licorice in blocks were out of specifications. Therefore samples of the two products of the last shipment were submitted for analyses:

- Licorice in blocks is slightly out of specifications as regards water and glycyrrhizic acid, but is nevertheless acceptable.
- Cocoa powder is identical with the first sample, this means largely out of specifications as regards the fat content (10 % instead of 20 - 26 %).

- Ingredients used in PMG Berlin (11)

Because of taste problems with the MLK-DB test cigarettes, the ingredients used were submitted to an analytical checking.

All the products were within specifications (invert sugar, glycerine, propylene glycol, chocolate liquor, ethanol) or were comparable with preliminary shipments (MFSBC Powder, Honey Flavour). Certain doubts about MFSB 1 - Liquid, as regards odour and GC-fingerprints, were eliminated after a local panel test in Munich.

- SFC 152 (PMG Munich) (12)

Concerning taste problems on PMS-cigarettes produced in February 1981, it was found that the base used for AC-solution easily separates into layers.

General information was given to users to mix well the contents of the drums containing the bases before use (13).

3.4.1. TRIALS WITH NEW SUPPLIES OF FILTER COMPONENTS

- Activated Charcoal (DEGUSSA, Germany), "DESOREX" (14)

The sample is largely out of specifications as regards CCl_4 - index.

- Polyethylene glycol 600 (EIGENMANN & VERONELLI, Milano; produced by CHEMISCHE WERKE HÜLS, Marl (Germany)) (14)

The molecular weight is within the specifications. However, the acidity and water content are slightly out of the specifications.

Another sample for a second analysis will be ordered directly from the producer (15).

3.4.2. QUALITY CONTROL OF FILTER COMPONENTS

- Triacetin for filter production of MLF-cigarettes in Bulgaria (16)

The sample corresponds to our specifications.

3.5.1. TRIALS WITH NEW SUPPLIES OF FILTER GLUES

- Liquid glue for KDF II (filter rod seam) (HENKEL & CIE AG, Pratteln (CH); "7693" and "7630"/yellow sheet 6057, 2 x 10 kg)

Machineability: The type "7630" was only applicable at reduced machine speed. After addition of 3 % water to the type "7693", filter plugs were produced at the maximum speed.

Subjective evaluation (only for the glue "7693"): A significant difference between the trial and the standard was found. The trial was rejected.

A further trial with another type from this producer is planned.

3.8.1. TRIALS WITH NEW SUPPLIES OF PACKING GLUES

- A MOLINS HL packing machine in Cousset was transformed to a higher speed (250 HLP/min). Because of gluing difficulties with the standard glue, a series of trials with alternative products was organized. When the trials were run the machine speed was at 235 HLP/min.
- "PLUS RAPIDE" (W. LAESSER, Erlinsbach/yellow sheet 6017, 25 kg)
Machineability: The glue is not better than the standard.
- "V 6" (W. LAESSER, Erlinsbach/yellow sheet 6027, 25 kg)
Machineability: The glue is not better than the standard.
- "V 3" (W. LAESSER, Erlinsbach/yellow sheet 6027, 25 kg)
Machineability: A significant improvement in comparison with the standard glue was found.
300 kg of this glue were ordered for a long-term test (May 11, 1981).
- Glue for the flap of the "export" case (W. LAESSER, Erlinsbach; "LESSO 1138" and "LESSO 1138-67" / yellow sheet 5974, 2 x 20 kg)
The glue is applied semi-automatically with a spray gun. "LESSO 1138-67" showed better properties than "LESSO 1138".
300 kg of the first were ordered for a long-term test.
- Glue for the flap of the "export" case (W. LAESSER, Erlinsbach; "LESSO 1138-67" / yellow sheet 5981, 300 kg)
The first results were confirmed. The glue has been accepted as a standard glue.

4.1.1. TRIALS ON PRE-CUTTING SOLUTIONS

- Artificial honey in MF-PC solution
An industrial trial on MLF-cigarettes with 100 % artificial honey was organized.
Smoke analyses: No significant difference between the standard and the trial was found.
Subjective evaluation: A significant difference between the trial and the standard was found. The trial was rejected. All the test cigarettes with 100 % artificial honey seemed to have the same defect: They "dried" the mouth.
Based on the result of the subjective evaluation, trials with different amounts of honey flavour on the basis of 100% artificial honey are planned.

5.1. PRODUCT QUALITY

- Spots on MLF-cigarettes (17)

In a normal production, spots on MLF-cigarettes were observed.

It was found that neither the humectant content nor the moisture content was irregular. Also the applied reconstituted sheets (RCB US and CH) were comparable with preliminary shipments.

As the spots were UV-active, they had been caused by a machine oil. By means of UV-spectra, the product was identified as an oil which is used on cigarette machines (MACOMA 150).

5.3. ASSISTANCE TO OTHER COUNTRIES

- Analyses for the ETNA-plant PMG Munich Humectants in tobacco (18 samples)

- Analyses for the ETNA-plant PMH Bergen op Zoom Humectants in tobacco (30 samples)

5.4. SERVICES FOR OTHER GROUPS

- Analyses for Process Development

SiO₂ in dust and tobacco (19 samples)

Chlorides in tobacco (11 samples)

Ash in tobacco (11 samples)

Calcium, Potassium and Magnesium in tobacco (11 samples)

- Analyses for Product Development

Plasticizer on filters by GC (13 samples)

- Analyses for Research Department

HWS in tobacco (6 samples)

Chlorides in solutions (45 samples)

REFERENCES

1. Report Schwarb-A (July 9 1981)
2. Letter from Schwarb-A to the Casing Kitchen (June 11 1981)
3. Report Schwarb-A, "Modèle MF-PC, fabriqué au laboratoire" (May 18 1981)
4. Report Schwarb-A "Contrôle de la qualité des sauces et parfums" (December 17 1980)
Report Schwarb-A "Contrôle de la qualité des sauces et parfums" (May 19 1981)

5. Minutes of the meeting "Qualité des sauces et parfums"
(December 18 1980)

Minutes of the meeting "Qualité des sauces et parfums"
(June 11 1981)

6. Telex from Widmer-A to Van Duuren-B (July 17 1981)
7. Letter from Widmer-A to Froideveaux-R (June 12 1981)
8. Letter from Widmer-A to Froideveaux-R (July 21 1981)
9. Letter from Widmer-A to Trento-A (May 21 1981)
10. Monthly report Widmer-A (February 1981)
Letter from Widmer-A to Trento-A (July 21 1981)
11. Letter from Widmer-A to Tessendorf-W (May 13 1981)
12. Letter from Schwarb-A to Lopes-F (March 24 1981)
Letter from Schwarb-A to Lopes-F (April 10 1981)
13. Letter from Lopes-F (March 17 1981)
Letter from Schwarb-A to Casing Kitchen (April 8 1981)
14. Letter from Widmer-A to Schembri-A (July 14 1981)
15. Letter from Widmer-A to Gmünder-R (July 14 1981)
16. Telex from Widmer-A to Lubomirski-J (May 25 1981)
17. Letter from Schwarb-A to Grossen-E (June 17 1981)



ALW/edk/JULY 31 1981

0000144254

PROJECT TITLE : SPECIFICATIONS
PERIOD COVERED : MAY 23 - July 24 1981
WRITTEN BY : Flury-C. (CAF)

1. FTR Switzerland (References 1 - 3)

- New complete specs were established for projects GOSTA 1 and GOSTA 2, products Bond 1 and Bond 4 for sale in Sweden (1).
- New complete specs were established for project TEXAS, cigarette Flint 3, for sale in Switzerland (2).
- New product version: PMM 044, Philip Morris Multifilter HL 85 mm, for sale in Cameroon.
- Modified processing and cigarette making specs for "ETNA step 2" introduction in seven blends (3).

2. PMH Holland (Reference 4)

- New specs were established for project BELTOISE, cigarette MAC Muratti Ambassador Extra Mild, for sale in Italy.
- New specs are available for project QUEEN, cigarette MED, for sale in Italy.
- New product version: MFM 090 Marlboro Menthol, for sale in Holland.
- New specs for the preparation of ETNA.

3. PMG Germany (References 5 - 6)

- Modified processing and cigarette making specs for ETNA (step 1) introduction in blends ML 010 Marlboro-IT and ML 012 Marlboro Menthol KS.
- Modified specs for PMS Philip Morris Super Lights (ETNA) content and filter paper)
- Packing spec for brand protection of SAG 040 Saratoga, for West Germany.

0000144255

4. LICENSEES - Ivory Coast

A material specs file has been prepared for the diluted MLF Marlboro Filter cigarette (with a 20-mm filter).

5. Material Specifications

The sections

TA/FA Ingredients/filter additives
FM Filtration material/tows
FP Filter paper
CP Cigarette paper
CB Cardboards
HL Hinge Lid blanks

of the "Usage of materials" file have been updated.

The following material specs were submitted to the suppliers:

- Rhodiaceta for tows 3,0/43'000 I and Y
- Baumgartner for filters Lark 126 and 132 mm and Bond 4
- Filtrona for filter Bond 1
- 11 suppliers for MLF Marlboro HL blank (Invercote D)

6. PME STANDARD RECIPES

Two new recipes, FLT-PC and BSE-AC, have been established.

7. Specifications on EDP

The cigarette making specs of all PME production centers are recorded in the Neuchâtel application, so that specs modifications can be calculated on computer in the future.

REFERENCES : 1-3 Specs FTR, Grossen-E., memos May 5, June 30, July 17, 1981
4 Specs PMH, memo July 6, 1981
5 Specs PMG, Buchmann-A., memos July 6, July 16, 1981

CAF/caf/JULY 24 1981

0000144256

PROJECT TITLE : PROCESS ASSURANCE
PERIOD COVERED : JUNE 1 - JULY 31 1981
WRITTEN BY : Bel-T. (THB)

1. AccuRay - Tobacco Weights (1 and 2)

Results of the first 3-month industrial test were presented to the PME Management. The following decisions have been taken:

- a) For the same brand with similar blend, the specified tobacco weight at 12 % should be the same between all production centers concerned.
- b) Each production center should have the possibility of choosing its maximum saving of tobacco, between 0 and 2,5 %.
- c) A limit of - 9 % of the tobacco weight for the reject point was considered too severe. A new trial should therefore be organized with - 9,5 %.

Following this conclusion, we decided to organize a second trial on the brand Marlboro only (MLF-MLK-MLH). The test will involve three production centers: Munich, Weltab, and FTR. Silvertown and Berlin will not be involved in this trial, as they work with a different blend. The trial in Bergen op Zoom will be postponed to the beginning of 1982 because of the start-up of the new primary in the period foreseen.

As the percentage of IS-ES-ET is different depending on the production centers, due to ET introduction, we fixed a density of 267 mg/ml for a Marlboro blend without IS-ES-ET.

All new tobacco weights have been calculated on this basis and sent to the production centers concerned. The lower limit cigarette rod has been fixed at - 9,5 % of the tobacco weight.

At the start of the trial, the lower mean weight limit will be
= 2,5 % for Munich and FTR
= 1,5 % for Weltab.

2. Non-Tobacco Material (3)

Based on the different trials made in the production centers, the following figures have been adopted as PME standards:

- Quantity of glue on the cigarette seam:
1,5 mg for all cigarette rod lengths.
- Quantity of glue on the tipping paper:
5,0 mg for a tipping length between 17 and 22 mm
7,0 mg " " " " " 23 " 28 mm
9,0 mg " " " " " 29 " 36 mm.

4. Dilution (4)

Due to the introduction of PDI/DDI values on the cigarette specifications, we fixed the figures to be adopted for brands produced in more than one production center. A range of values will be specified from now on.

14. ETNA (5)

Following the first trial made in Silvertown in January, which did not give very satisfactory results, a second test was organized in June. We found an average of 8,0 ET in the cigarettes (specification: 10 %).

15. Quality Workshop (6 and 7)

The monthly report supplements covering the months April and May have been sent to PME Management and to the QA managers. After a period of action of 3 months, we can say that a good improvement may be particularly noted in the section "packet".

16. MTI Verona and Rovereto (8)

Following our one-week visit to these two factories, the final report has been distributed.

18. Reduction in Tobacco Weights (9)

Due to some incorrect calculations which occurred in some production centers when introducing ETNA, instructions have been issued to all factories to use the right correction factors for improved stems IS, expanded stems ES, and expanded tobacco ET.

- REFERENCES : 1. Standardized tobacco weights and AccuRay limits
Bel-T memo to J.B. Boder (June 1981).
2. Marlboro Standardized Tobacco weights and AccuRay limits, 2nd trial
Bel-T memo to Q.A. Managers (July 1981).
3. Weight of glue on cigarette
Bel-T memo to Q.A. Managers (July 1981).
4. Dilution-US figures on specifications
Bel-T memo to Q.A. Managers (July 1981).
5. Addition of Etna to Marlboro UK blend in PM Silvertown
Bel-T to A. Read (July 1981).
6. Quality Workshop, Monthly Report Supplement (June 1981).
7. Quality Workshop, Monthly Report Supplement (July 1981).
8. Rapport de visite Verona + Rovereto (June 1981).
9. Reduction of Tobacco weights when introducing Expanded Tobacco
Bel-T memo to Q.A. Managers (July 1981).
- T. Ry

THB/amk/AUGUST 3 1981

0000144259

PROJECT TITLE : INSTRUMENTATION AND PROCESS
AUTOMATION

PERIOD COVERED : JUNE 23 - JULY 22

WRITTEN BY : Thévoz-M. (MIT)

Automation of the Smoking Laboratory (Project COLDAC)

The authorization concerning the request for investment was granted on July 6 1981. The computer and terminals to be used in this project will be delivered between 15 and 30 October 1981. The training course on the material and software will be given by Hewlett-Packard between September 20 and December 20 1981.

The specifications of this application are currently being studied by the various users. A technical notebook containing all the information concerning the smoking laboratory's instrument link-up is now being written by the Instrumentation group. The setting-up of the "data link" in the laboratories is planned to be carried out in September 1981. The final positioning of the terminals and the allocation of work places is still being prepared. A series of 65 junction boxes will be installed during the setting-up of the link-up to give maximum flexibility in the geographical distribution of work places.

RTD/DIL Data Treatment

A mini HP 85-type computer will be set up in the Quality Control laboratories of the Serrières production center. This special computer will perform the daily and monthly treatment of RTD/DIL data.

This treatment work consists of watching over, actively and interactively, the differences between the values measured and the specifications of each brand. The mini-computer produces on a print-out the values recorded during the day for each brand by each machine. The results are stored on magnetic tape and will be used to establish monthly and quaterly reports.

The specifications of this application were discussed in detail with the various users and heads of Quality Assurance. An evaluation programme has been written in BASIC for the daily treatment of data and will be tested in the coming months. The characteristics of this programme will be given in the next monthly report.

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The direct link-up of the RTD/DIL measuring instrument (SODIM-type) to the HP 85 micro-computer is currently being studied. This connection will enable us to do away with the manual introduction of numerical data on a keyboard which is a particularly long and fastidious operation. This on-line connection is planned for autumn 1981.

Reilly

MIT/jud/JULY 30 1981

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0000144261

PROJECT TITLE : PATENTS
PERIOD COVERED : May 17 - July 16, 1981
WRITTEN BY : Mandiratta-J-C (MJA)

PROJECT EXIT

It was decided during a meeting attended also by Mr. Arthur Palmer at Reddie & Grose's office (our Patent Agents in England) on June 29, 1981 to lodge the poor man's opposition in the Dutch and Swedish Patent Offices. Instructions to do so have been given to Dr. Hach who had submitted a similar opposition in the West German Patent Office. It was also agreed not to lodge such an opposition in the French Patent Office because according to the French patent law the same will not be considered by the examiner at this stage.

CANDIDA "BYPASS" NEW APPLICATION

Abstracts of this application were sent to the patent department in Richmond. The final agreement has been reached now to file the priority application in West Germany. This application is being prepared and will be filed shortly after the inventor's approval.

NEW APPLICATIONS

Details of our new cases 28234, 28237, 28243, 28244 and 28247 have been given to Reddie & Grose who will now proceed to filing these cases in different countries as recommended by the PME Patent Committee via EPO or EPC. Prior art is being prepared for these cases and will be sent shortly along with the priority documents to the patent department, Richmond, and to Reddie & Grose.

WEST GERMAN OFFENLEGUNGSSCHRIFT 2942544
ASSIGNEE: TKR Dritte Tabak Forschungs GmbH, Munich
Published on April 30, 1981

In this application urea is claimed as additive for reducing harmful substances in tobacco smoke. Prior art has been sent to Reddie & Grose and we are waiting for them to inform us as to whether this application should be opposed.

PATENT DOCUMENTATION

To date, 8500 documents are on STAIRS system for patent documentation.



MJA/mle/July 16, 1981

PROJECT TITLE : LEGISLATION
PERIOD COVERED : JULY 1981
WRITTEN BY : Fink-W. (WAF)

COUMARIN

On April 1, 1981, Reemtsma (Hamburg) was granted an exceptional permission to use coumarin on tobacco in West Germany (BMJFG of March 25, 1981 / 414-6380-4/41). The use of coumarin is limited to 100 ppm/cigarette for a total of 10 billion cigarettes p.a. The exceptional permission has been given for the period of April 1, 1981 to March 31, 1983.

CONTAMINATIVE ORDINANCE ("Kontaminanten-Verordnung")

The German Federal Government is currently preparing a "Contaminative Ordinance". The ordinance, to be issued at the end of 1981, will establish the maximum permissible amounts of the metals lead, mercury and cadmium on and in foodstuff. It is not yet clear as to the extent to which tobacco and tobacco products are affected by this regulation.

W. Fink

WAF/jiq AUGUST 3 1981

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T. S. OSDENE

AUG 24 1981

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